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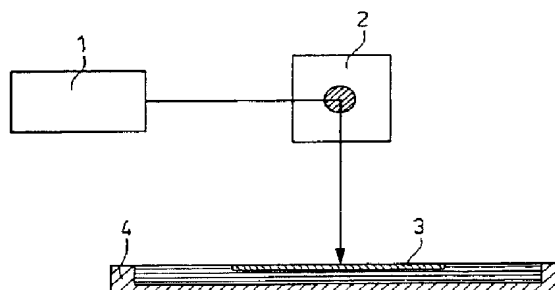
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(54) **PROCEDE ET MATERIAU POUR LA FABRICATION DE CORPS SERVANT DE MODELES**

(54) **METHOD AND MATERIAL FOR PRODUCING MODEL BODIES**

(57)

Model bodies of any form or shape can be produced from special plastic powders with the aid of selective sintering using infrared lasers. Also described are plastic powders containing an infrared absorber and suitable for laser- assisted model production.





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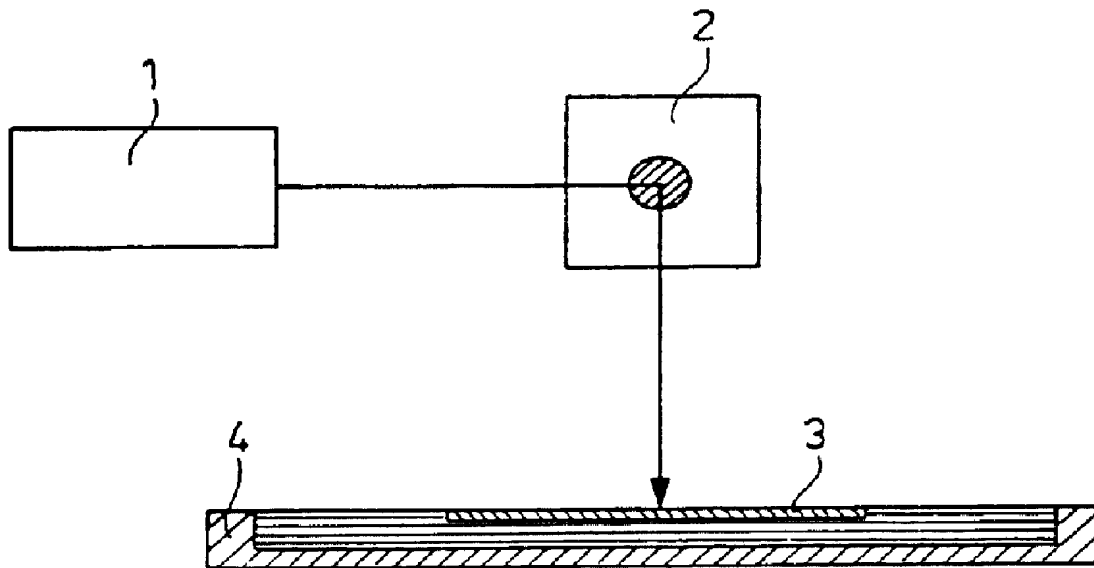
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(57) **Abrégé/Abstract:**

Model bodies of any form or shape can be produced from special plastic powders with the aid of selective sintering using infrared lasers. Also described are plastic powders containing an infrared absorber and suitable for laser-assisted model production.

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(57) Abstract

Model bodies of any form or shape can be produced from special plastic powders with the aid of selective sintering using infrared lasers. Also described are plastic powders containing an infrared absorber and suitable for laser-assisted model production.

**A process and a material for producing model bodies**

This invention relates to a process for producing model bodies, wherein, using plastics in the form of selected plastics powders, an arbitrary three-dimensional structure can be built up with the aid of selective sintering using laser light in the IR range. The invention also relates to a special plastics powder which contains an IR absorber and which is particularly suitable for sintering with IR laser light.

The invention relates in particular to a process for producing three-dimensional models from plastics in accordance with stored, geometric data with the aid of a computer-aided installation which operates using IR laser beams for the direct production of prototypes and models (a rapid prototyping installation).

The term "rapid prototyping" summarises the computer-aided, additive, automated methods of constructing models which are currently known. The term "laser sintering" denotes a rapid prototyping process in which a loose pile of defined powdered materials can be heated and sintered at defined points on planes under the action of laser beams which are preferably controlled by a program.

The use of plastics powders for laser sintering by means of CO<sub>2</sub> lasers is known (A. Gebhardt, Rapid Prototyping, Carl Hanser Verlag, Munich, Vienna 1996, pages 115-116). A process is described there for the production of model bodies in which an arbitrary three-dimensional structure can be built up by selective sintering using plastics, with the aid of light from a CO<sub>2</sub> laser.

One disadvantage of processes known hitherto is the limited accuracy of the mouldings which are obtained. Due to this lack of accuracy, mouldings which are currently produced in this manner have to be subjected in many cases to costly manual re-working. The low degree of accuracy is partly a consequence of the CO<sub>2</sub> laser used which has a wavelength of 10.6  $\mu\text{m}$  and which can only be poorly focused. Lasers with improved focusing, such as the Nd-YAG laser with a wavelength of 1064 nm, have not

hitherto been used for laser sintering, since customary plastics do not absorb at this wavelength.

5 The present invention relates to a process for producing three-dimensional models from plastics in accordance with stored, geometric data with the aid of laser beams, which are controlled according to these data and which have a wavelength of 500 to 1500 nm, preferably 800 to 1200 nm, wherein laser beams are directed corresponding to the geometric data on to defined three-dimensional zones of a poured bed of a fine particulate plastics powder, and the material is fused or sintered, characterised in that  
10 the plastics powder has an average particle size of 2 to 200  $\mu\text{m}$  and contains an IR absorber.

In one particular embodiment of the present invention, the plastics powder is substantially spherical.

15 The present invention also relates to plastics powders for use as a starting material for the laser-assisted production of models, wherein the powder particles have an average particle size (i.e. the weight average of their diameter) of 2 to 200  $\mu\text{m}$ , and contain an IR absorber.

20 Different types of lasers are suitable for the process according to the invention. Solid lasers and semiconductor diode lasers are particularly suitable. Examples of solid places include Nd-YAG lasers with a wavelength of 1064 nm, and Nd-YLF lasers with a wavelength of 1053 nm. Suitable diode lasers are those which emit at 823 nm or 985  
25 nm.

During irradiation, the energy which is irradiated on to the surface of the poured poile of powder preferably ranges from 0.01 to 100  $\text{mJ}/\text{mm}^2$ , most preferably from 1 to 50  $\text{mJ}/\text{mm}^2$ .

30 Depending on the application, the effective diameter of the laser beam preferably ranges from 0.001 to 0.05 mm, most preferably from 0.01 to 0.05 mm.

Pulsed lasers are preferably used, and a high pulse frequency, particularly from 1 to 100 kHz, has proved to be particularly suitable.

5 The preferred procedure can be described as follows:

the laser beam impinges on the uppermost layer of the poured bed of the material used according to the invention and in so doing fuses or sinters the material down to a defined layer thickness. This layer thickness can range from 0.005 mm to 1 mm, preferably from 0.01 mm to 0.5 mm.

10

In this manner, the first layer of the desired component is produced. The working volume is subsequently reduced by an amount which is less than the thickness of the sintered layer. The working volume is filled with additional polymer material up to the original height. The second layer of the component is sintered by repeated irradiation  
15 with the laser, and is bonded to the preceding layer. The further layers which are required in order to finish the component are produced by repeating the procedure.

The laser beam is used at a velocity of 1 to 1000 mm/s, preferably 10 to 100 mm/s.

20 Plastics powders which are suitable for the invention can belong to different classes of polymers. Examples thereof include: polyolefines such as polyethylene and polypropylene, polyamides such as polyamide-6 and polyamide-6,6, polyesters as such as polyethylene terephthalate, polycarbonates, fusable polyurethanes, polystyrene, styrene-acrylonitrile copolymers and polyacrylates. Plastics powders based on partially  
25 crystalline polymers are particularly suitable for the production of pore-free model bodies.

The particle size of the powder particles is particularly important for the process according to the invention. In general, the average particle diameter ranges from 2 to  
30 200  $\mu\text{m}$ , preferably 5 to 100  $\mu\text{m}$ , most preferably 5 to 50  $\mu\text{m}$ . The average particle diameter (particle size) which is quoted here means the weight average diameter.

In order to produce the particle size which is essential for the process, plastics which usually exist as coarse granules can be ground. Under some circumstances, however, this produces an edged or angular shape of the plastics particles. Particles such as these, with their irregular or uneven surfaces, sometimes exhibit poor flow properties, which have a disadvantageous effect on the processing thereof in laser sintering installations. Flow enhancing agents therefore usually have to be added to the plastics in order to improve the flowability of the comminuted plastics and to ensure that automated installations can be operated therewith.

10 Polymers which are substantially spherical in shape (pearl polymers) are particularly suitable for the process according to the invention.

It has been shown that pearl polymers are also suitable for the laser sintering process according to the invention. Moreover, they exhibit flow properties which are much more favourable than those of other, ground plastics, and therefore do not even necessitate the addition of flow enhancing agents in order to improve their flow properties.

A further significant advantage of pearl polymers is that on incineration, e.g. as the nucleus of a hollow ceramic moulding, they do not leave behind any troublesome residues. In the case of ground plastics particles to which flow enhancing agents are added, it has been observed that they cannot be incinerated without leaving a residue.

This is particularly important if the models made of plastics which are primarily produced by means of laser sintering are further processed in subsequent process steps to produce a fine casting. For this purpose, for example, after the model produced by the process according to the invention has been coated with wax in order further to improve the surface of the model, the model is immersed in a slurried ceramic material, and the model which is coated with ceramic material is calcined in a furnace. The model should burn away completely during firing, leaving the exposed hollow moulding made of ceramic. Since conventional ground plastics do not decompose

completely on combustion due to the added flow enhancing agents, metallic models which are subsequently cast in the ceramic mould often exhibit surface inaccuracies.

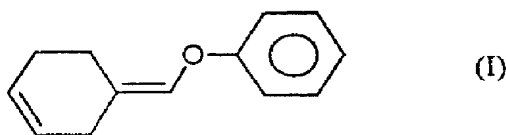
5 A further advantage of the use of pearl polymers arises with regard to the surface accuracy and surface roughness of the models which are produced by the process according to the invention. On account of their round shape and their good flow properties, models which are produced using the preferred pearl polymers are smoother and are thus more accurate also.

10 The pearl polymers preferably consist of homo- or copolymers of mono-ethylenically unsaturated compounds (monomers). Copolymers in the sense of the invention are to be understood to be polymers which are synthesised from two or more different monomers. Examples of suitable monomers include styrene, alpha-methylstyrene, chlorostyrene, esters of acrylic acid such as ethyl acrylate, butyl acrylate, 2-ethylhexyl  
15 acrylate, decyl acrylate, dodecyl acrylate, esters of methacrylic acid such as methyl methacrylate, ethyl methacrylate, isopropyl methacrylate, n-butyl methacrylate, isobutyl methacrylate, hexyl methacrylate, 2-ethylhexyl methacrylate, decyl methacrylate, dodecyl methacrylate and stearyl methacrylate, as well as acrylonitrile, methacrylonitrile, methacrylamide and vinyl acetate.

20

It has been shown that the molecular weight of the pearl polymers is important as regards the suitability thereof for the process according to the invention. In particular, the molecular weight (weight average, Mw) should preferably range from 10,000 to 500,000, most preferably from 20,000 to 250,000 daltons. Molecular weight regulators  
25 can be used during the production of the pearl polymers in order to attain the desired molecular weight. Molecular weight regulators which are particularly suitable include sulphur compounds, e.g. n-butyl mercaptans, dodecyl mercaptan, ethyl thioglycolate and diisopropylxanthogen disulphide. The regulators cited in DE 3 010 373 are also very suitable for adjusting the molecular weight, for example the enol ether  
30 corresponding to formula I





Particularly suitable pearl polymers can be produced by known methods. Thus pearl polymers with a particle size of about 10 to 200  $\mu\text{m}$  can be obtained by suspension polymerisation or pearl polymerisation. The term "suspension polymerisation" is to be understood to mean a process in which a monomer or a monomer-containing mixture, which contains an initiator which is soluble in the monomer(s), is comminuted in the form of droplets, optionally in admixture with small, solid particles, in a substantially immiscible phase which contains a dispersing agent, and is cured by increasing the temperature and stirring. Further details of suspension polymerisation are described, for example, in Ullmanns Encyclopedia of Industrial Chemistry, Volume A21, 5<sup>th</sup> Edition, VCH, Weinheim 1992, on pages 363 to 373.

Pearl polymers with particle sizes of 2 to 10  $\mu\text{m}$  can be produced by what is termed dispersion polymerisation. In dispersion polymerisation, a solvent is used in which the monomers employed are soluble but in which the polymer formed is insoluble. Dispersion polymerisation generally produces pearl polymers with a narrow particle size distribution.

In principle, all compounds which absorb light at a wavelength from 500 to 1500 nm, preferably 800 to 1200 nm, are suitable as IR absorbers. Both IR pigments and IR dyes can be used independently of each other.

Carbon black, particularly synthetically produced carbon black, is preferably used as an IR pigment. The carbon black which is used preferably has a specific surface of 10 to 500  $\text{m}^2/\text{g}$ , as measured by the BET method. Suitable types of carbon black include channel blacks, furnace blacks and flame-produced lampblacks.

Moreover, mixed metal oxide pigments of the rutile or spinel type are suitable.

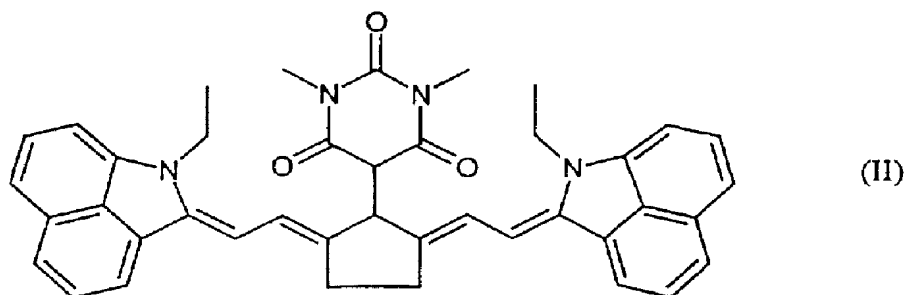
Examples of suitable metal oxide pigments include the commercially available products HEUCODUR<sup>®</sup>-Brown 859 and HEUCODUR<sup>®</sup>- Black 953.

IR dyes (infra-red absorbing dyes, IRDs) are known in the art. IR dyes from various  
5 classes of substances are suitable, e.g. indoaniline dyes, oxonol dyes, porphine derivatives, anthraquinone dyes, mesostyryl dyes, pyrilium compounds and squarylium derivatives.

IR dyes according to DE-OS 4 331 62 are also particularly suitable, since they have a  
10 narrow absorption in the visible region and thus make it possible to produce 3D models by the process according to the invention which are not coloured or which are only slightly coloured.

The IR dye corresponding to formula II can be cited as an example:

15



According to the invention, the amount of IR absorber ranges from 0.01 to 10 % by weight, preferably from 0.05 to 5 % by weight, with respect to the plastics powder.

20

The production of plastics powders which contain an IR absorber can be effected in various ways. Thus it is possible to mix the plastics material with the IR absorber in the melt with the aid of an extruder and to comminute the extrudate which is obtained to the desired particle size in a mill. It is also possible to add the IR absorber during the  
25 production of the plastics material, so that the IR absorber is incorporated in the plastics material which is formed. During the production of pearl polymers by suspension polymerisation, the IR absorber can be added to the monomers.

It has been found that plastics particles can be doped in a very simple manner with soluble IR dyes. In this process, the plastics particles are dispersed in a liquid phase, preferably water, which does not dissolve the plastics material, and a wetting agent or surfactant can also be used. Examples of suitable surfactants for this application include sodium alkylsulphonates, sodium sulphosuccinic acid isooctyl esters and ethoxylated nonylphenol. A solution of the IR dye is added to the dispersion which is obtained, wherein a solvent which is preferably immiscible with water can be added, e.g. ethyl acetate, toluene, butanone, chloroform, dichloroethane or methyl isobutyl ether. During this treatment, the solvent, including the IR dye, swells into the plastics particles. The water can subsequently be removed by filtration or decantation, and the solvent can be removed by evaporation, e.g. under reduced pressure, whereupon the IR dye remains in the plastics particles.

The plastics powders according to the invention, which contain an IR absorber, are particularly suitable for the laser sintering process using IR lasers, particularly Nd-YAG lasers, and provide models or components with particularly good accuracy of detail.

Figure 1 is a schematic illustration of a rapid prototyping installation.

The following examples illustrate the production of finely divided plastics materials and describe experiments on the sintering of these materials with the aid of an IR laser.

**Example 1**

Production of a pearl polymer according to the invention which contained carbon black

a) Carbon black dispersion

5

6 g carbon black (Printex G supplied by Degussa, BET specific surface 30 m<sup>2</sup>/g), 24 g polymethyl methacrylate and 216 g methyl methacrylate were treated for 2 hours in a ball mill, whereupon a homogeneous dispersion was obtained which did not settle out.

10 b) Pearl polymer

200 g of the dispersion from a) and 2.0 g of 2,2'-azobis(isobutyronitrile) were intensively mixed. The mixture was transferred to a stirred reactor which had previously been filled with 1.0 litre of a 1 % by weight, aqueous alkaline solution of a copolymer of 50 % by weight methacrylic acid and 50 % by weight methyl  
15 methacrylate, the pH of which solution had been adjusted to pH 8 with sodium hydroxide solution. The stirrer speed was set at 700 revolutions per minute, and the temperature was maintained at 60°C for 3 hours, then at 78°C for 10 hours, and then at 85°C for 2 hours, followed by cooling to room temperature. The pearl polymer which was formed was isolated by decantation, washed repeatedly with water and dried at  
20 50°C under vacuum. 168 g of a pearl polymer with an intensive black coloration was obtained, which had an average particle size of 18 µm and a molecular weight Mw of 230.000.

**Example 2**

25 Production of a pearl polymer according to the invention which contained carbon black

a) carbon black dispersion

12 g carbon black (Printex G supplied by Degussa), 28.8 g polymethyl methacrylate,  
30 216 g methyl methacrylate and 43.2 g n-butyl methacrylate were treated for 2 hours in a ball mill, whereupon a homogeneous dispersion was obtained which did not settle out.

**b) Pearl polymer**

250 g of carbon black dispersion a) and 2.5 g 2,2'-azobis(isobutyronitrile) were intensively mixed. The mixture was transferred to a stirred reactor which had previously been filled with 1.25 litres of a 1 % by weight, aqueous alkaline solution of  
5 a copolymer of 50 % by weight methacrylic acid and 50 % by weight methyl methacrylate, the pH of which solution had been adjusted to pH 8 with sodium hydroxide solution. The stirrer speed was set at 600 revolutions per minute, and the temperature was maintained 78°C for 10 hours, and then at 85°C for 2 hours, followed by cooling to room temperature over 2 hours. The pearl polymer which was formed was  
10 isolated by decantation, washed repeatedly with water and dried at 50°C under vacuum. 205 g of a pearl polymer with an intensive black coloration was obtained, which had an average particle size of 25 µm and a molecular weight Mw of 220,000.

**Example 3**

15 Production of a pearl polymer according to the invention which contained an IR dye

**a) Production of the pearl polymer**

2340 g methanol, 180 g polyvinylpyrrolidone, 210 g methyl methacrylate and 90 g ethyl methacrylate were mixed to form a homogeneous solution in a 4 litre reactor  
20 fitted with a grid stirrer. This solution was heated over one hour to 55°C under nitrogen and at a stirrer speed of 100 rpm. A solution of 6 g 2,2'-azobis(isobutyronitrile) in 165 g methanol was then added to the reactor. The polymerisation mixture was stirred for a further 20 hours at 55°C and 100 rpm. The final polymer dispersion was subsequently cooled to room temperature and the pearl polymer was isolated by sedimentation. 193 g  
25 of a pearl polymer were obtained which had a molecular weight Mw of 75,000, an average particle size of 12 µm and an  $\bar{\phi}(90)/\bar{\phi}(10)$  value of 1.18. The ratio of the 90% value ( $\bar{\phi}(90)$ ) to the 10% value ( $\bar{\phi}(10)$ ) of the distribution by volume, namely the  $\bar{\phi}(90)/\bar{\phi}(10)$  value, is a good measure of the width of the distribution.  $\bar{\phi}(90)/\bar{\phi}(10)$  distributions less than 2.0 imply narrow particle size distributions.

b) Doping the pearl polymer with IR dye

100 g of the pearl polymer from a) were dispersed in a solution of 900g water and 200 mg sodium sulphosuccinic acid isooctyl ester. A solution of 284 mg of an IR dye of formula II and 100 g ethyl acetate were added drop-wise to this solution with stirring.

5 The mixture was stirred for 4 hours at room temperature and was subsequently treated with ultrasound for 10 minutes. The pearl polymer was filtered off, washed repeatedly with water and was dried to constant weight at 50°C under vacuum in order completely to remove the ethyl acetate.

#### 10 **Example 4**

Production of a pearl polymer according to the invention which contained an IR dye

a) Production of the pearl polymer

2500 g methanol, 64 g polyvinylpyrrolidone, 240 g styrene and 60 g ethyl methacrylate  
15 were mixed to form a homogeneous solution in a 4 litre reactor fitted with a grid stirrer. This solution was heated over one hour to 70°C under nitrogen and at a stirrer speed of 100 rpm. A solution of 3.75 g 2,2'-azobis(isobutyronitrile) in 75 g styrene was then added to the reactor. The polymerisation mixture was stirred for a further 15 hours at 70°C and 100 rpm. The final polymer dispersion was subsequently cooled to room  
20 temperature and the pearl polymer was isolated by sedimentation. 247 g of a pearl polymer was obtained which had an average particle size of 14 µm, a Ø (90)/Ø (10) value of 1.6, and a molecular weight Mw of 60,000.

b) Doping the pearl polymer with an IR dye

25 10 g of the pearl polymer from a) were dispersed in a solution of 90 g water and 20 mg sodium sulphosuccinic acid isooctyl ester. A solution of 28.4 mg of an IR dye of formula II and 10 g ethyl acetate were added drop-wise to this solution with stirring. The mixture was stirred for 4 hours at room temperature. The pearl polymer was filtered off, washed repeatedly with water and was dried to constant weight at 50°C  
30 under vacuum in order completely to remove the ethyl acetate.

#### **Example 5**

Sintering tests on the pearl polymers from Examples 1 to 4

20 g of the pearl polymer from Examples 1 to 4 were introduced into the supply vessel of an experimental installation as illustrated in Figure 1.

5

The beam of an Nd-YAG laser 1 (effective cross-section  $5 \text{ mm}^2$ , pulse frequency 10 Hz) was directed via the deflection mirror 2 at a velocity of 10 mm/s on to the surface of the poured bed 3 of pearl polymer and an area of  $20 \times 20 \text{ mm}$  was scanned by the laser beam. The irradiated energy corresponded to  $40 \text{ mJ/mm}^2$ . Rigid mouldings were

10

obtained in this manner from the pearl polymers from Examples 1b), 2b), 3b) and 4b).

The test specimens were mechanically broken in liquid nitrogen and the fracture surfaces were investigated by scanning electron microscopy. In the case of the pearl polymer from Example 3a) (comparative test without IR absorber), the product remained unchanged as a powder.

15

Pearl polymer from Example		Formation of a moulding	Surface	Assessment of fracture surface
1b)	according to the invention	+	smooth	smooth with pores
2b)	according to the invention	+	smooth	smooth with pores
3b)	according to the invention	+	smooth, glossy	smooth, pore-free
4b)	according to the invention	+	smooth, glossy	smooth, pore-free
3a)	comparison	-	-	-

**Claims**

1. A process for producing three-dimensional models from plastics in accordance with stored, geometric data with the aid of laser beams of wavelength 500 to 1500 nm, preferably 800 to 1200 nm, which are controlled according to said data, wherein laser beams corresponding to the geometric data are directed on to defined three-dimensional zones of a poured bed of a fine-grained plastics powder and the material is fused or sintered, characterised in that the plastics powder has an average particle diameter of 2 to 200  $\mu\text{m}$  and contains an IR absorber.
2. A process according to claim 1, characterised in that the plastics powder is a pearl polymer.
3. A process according to claims 1 or 2, characterised in that a solid laser, particularly a Nd-YAG laser with a wavelength of 1064 nm or a Nd-YLF laser with a wavelength of 1053 nm, is used as a laser or that a semiconductor diode laser, particularly one which emits at 823 nm or 985 nm, is used as a laser.
4. A process according to any one of claims 1 to 3, characterised in that during irradiation the energy density on the surface of the poured bed of powder ranges from 0.01 to 100  $\text{mJ}/\text{mm}^2$ , preferably from 1 to 50  $\text{mJ}/\text{mm}^2$ .
5. A process according to any one of claims 1 to 4, characterised in that the effective diameter of the laser beam ranges from 0.001 to 0.05 mm, preferably from 0.01 to 0.05 mm.
6. A process according to any one of claims 1 to 4, characterised in that a pulsed laser is used, wherein the laser has a pulse frequency of 1 to 100 kHz.



7.     Plastics powders for use as a starting material for the laser-assisted production of models, wherein the powder particles have an average particle diameter of 2 to 200  $\mu\text{m}$  and contain an IR absorber.
- 5     8.     Plastics powders according to claim 7, characterised in that the plastics powders consist of pearl polymers.
9.     Plastics powders according to claims 7 or 8, characterised in that they contain compounds which absorb light at a wavelength of 500 to 1500 nm, preferably 10     800 to 1200 nm, particularly IR pigments and/or IR dyes, as IR absorbers.
10.    Plastics powders according to claim 9, characterised in that they contain carbon black as an IR pigment, or contain a dye from the series comprising indoaniline dyes, oxonol dyes, porphine derivatives, anthraquinone dyes, mesostyryl dyes, 15     pyrilium compounds and squarylium derivatives as IR dyes.
11.    Plastics powders according to any one of claims 7 to 10, characterised in that they contain IR absorbers in an amount of 0.01 to 10 % by weight, preferably 0.05 to 5 % by weight, with respect to the plastics powder.
- 20    12.    A process for producing a plastics powder which contains an IR dye, characterised in that an aqueous dispersion of a plastics powder is brought into contact with a solution of an IR dye in a solvent which is immiscible with water and the water and the solvent are subsequently removed.

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**Fig. 1**

